CHAPTER 1

INTRODUCTION

1.1 PROJECT BACKGROUND

Intumescent coating is used to provide fire protection to the structural steel. The char structure provides a shield that insulates the metal substrate from radiant heat and direct contact with flame. However, heat and fire may transfer to the metal substrate through the cracks in char structure. The char structure formed from the coating is easily oxidized at high temperature [1].

Under the action of heat, intumescent fire retardant coatings will expand and form a thick porous charred layer to insulate the substrate. The substrate will be protected against high temperature rise and exposure to oxygen within a period of time. Applying this type of coatings is one of the most efficient ways of providing fire retardancy to flammable materials [2].

1.2 PROBLEM STATEMENT

Due to the poor thermal properties of Intumescent Fire Retardant Coating, the coating oxidizes at high temperature and looses their fire protection properties [1]. The lowest size of the coating with low expand volume is too low to give a significant performance in fire protection properties of the coating.

1.3 OBJECTIVE AND SCOPE OF STUDY

1.3.1 Objective

- To develop the Intumescent Fire Retardant Coating by adding Silica Sand as filler.

1.3.2 Scope of Study

a) Research on the formulation of Intumescent Fire Retardant Coating from various sources such as internet, articles, books and journals.

b) Develop a formulation based on the information gained.

c) Develop the intumescent coating with expandable graphite and coating with expandable graphite and silica sand based on the formulations.

d) The coating properties regarding the char layer will be analyzed based on the samples formulated by using Fire Test.

e) The sample produced will be tested in the laboratory by using SEM (Scanning Electron Microscopy) and XRF (X-Ray Fluorescence) technique.

f) An analysis will be done based on the result collected form the test.

1.3.3 Limiting of the Project

- Focusing on locally available silica sand.
- Develop the coating by using 63 μm particles size of expandable graphite, Melamine, Ammonium Polyphosphate, APP, Boric Acid, Silica Sand, Hardener and Epoxy Resin.
- Develop only 14 formulations for coating with expandable graphite and 8 formulations for coating with expandable graphite and silica sand.

CHAPTER 2

LITERATURE REVIEW

2.1 THE DEFINITON OF INTUMESCENT COATING

Intumescences may be defined as “thermally induced expansion of material” [7]. Intumescences also defined as the ability of the coating, upon exposure to high temperature flame, to swell or foam into a solid heat insulating layer while protecting the substrate from direct exposure to the flame. The coating retains its expanded or foamed structure at high temperatures to provide a heat isolative layer which protects the substrate for a prolonged period [5].

Intumescent coating system is a fire protection system applied to structural steel. Intumescent coatings are thin films product which is inert at low temperatures. The coatings swell (insulate the substrate) or intumesce in a controlled manner to many times its original thickness to produce a carbonaceous charred layer of low conductivity foam when exposed to high temperatures [5]. This char layers reduce the rate of temperature rise in the steel. This char also acts as a physical barrier against heat transfer to the surface of combustible material.

2.2 CLASSIFICATION AND MECHANISM OF INTUMESCENT COATING

There are two distinctly different types of intumescent coatings. The first one is the traditional chemical intumescent fire retardant coatings with chemical fire retardant mechanism. This type of coating consists of three basic components which are a carbon source (pentaerythritol), an acid source (Ammonium Phosphate), and a gas source (nitrogen). Ideal compatibility between these three basic components is essential to producing an excellent fire retardancy, that is, dehydration to char and

release of gas in a transitional semi-liquid state sufficient to enable foam and expansion to occur, followed by full carbonization without char collapse [2].

The second one is a new type of physical intumescent coatings containing expandable graphite with physical fire retardant mechanism. Expandable graphite is formed by treating crystalline graphite, which is composed of stacks of parallel planes of carbon atoms, with intercalants such as sulfuric acid or nitric acid. The development of these coatings is focused on improving the char strength of the intumescent fire retardant coating [2].

2.3 BASIC ELEMENTS IN THE INTUMESCENT COATING

Intumescent coatings contain four basic elements which are [4]:

- Acid source or catalyst; a dehydrating or carbonizing agent, such as Ammonium Polyphosphate (APP), which at temperatures above 200^0C liberates polyphosphoric acid.
- Carbon source; organic substances which can be charred and turned into coal by polyphosphoric acid such as pentaerythritol or dipentaerythritol.
- Blowing agent such as melamine, which under decomposition release gases (N_2, NH_3) and expands the char.
- Binder; such as epoxy resin makes the compounds contact each other.

2.4 THE INTUMESCENT COATING PROCESS

There are four steps involve in the coating process which are [7]:

a) The heat begins to soften the polymeric binder. The heat also causes a release of an organic acid (from Ammonia Polyphosphate).
b) Carbonization of the polyols begins to take place.

c) Gas is then produced through the decomposition of the blowing agent (Melamine), swelling the molten mixture.
d) Solidification of the foamed char, through a cross-linking reaction to maintain the insulation.

In an ideal situation, intumescent can expand to around 100 times its original thickness. To achieve this requires careful selection of formulation components and precise matching of processes involved.

2.5 SILICA SAND AS FILLER

Silica sand is used as filler in the intumescent coating to protect the steel substrate. Coating possesses unusual fire-protective properties that are due to two mechanical effects which are intumescence and evaporation. In the heat of a fire, the coating will expand with water vapor, forming a heat-insulating layer. This process is known as intumescence. The coating can expand to 300%-500% of its initial thickness [1]. This coating can retain its intumesced structure for prolonged period at temperatures up to 1000^0C [5].

In addition, heat energy is absorbed by the water in the coating both when vaporizing during intumescence and when leaving (evaporating from) the expanded coating. This serves to cool the surface. Together, these effects form an excellent fire-protective performance by the coating. The coating remains chemically stable at extreme temperatures, and is capable of meeting the highest fire safety and environmental standards [8].

2.6 WHAT IS EXPANDABLE GRAPHITE?

Expandable graphite is manufactured by using natural graphite flake. Natural graphite flake is a layer crystals consists of sheets of carbon atoms. The atoms are tightly bound to each other within a layer, but the layer themselves are weakly held together [9].

Expandable graphite is manufactured by the oxidation of graphite flake in sulphuric acid. The capillary structure of graphite flake permits them to absorb the acid readily. Intercalated graphite when heated at high temperature it swells or expands up about 100 times to its original volume. The origin of expansion lies in the vaporization of intercalates. The expanded graphite flakes are vermiform in appearance and have a circumference of half inch and length of sometimes several inches. The volume of this "worm" or char structure is often a hundred or more times than that of the original graphite [9].

2.7 HOW DOES EXPANDABLE GRAPHITE WORKS AS FLAME RETARDANTS?

When expandable graphite exposed to heat, it expands to more than 100 times to its original volume and covers the entire burning surface by "worm" like a structure of expandable graphite. Expandable graphite act as a char former, as insulating agent due to the formation of small air gaps between graphite layers. It will reduce the heat release, mass loss, smoke generation and toxic gas emission [9].

The expansion of expandable graphite must occur at critical temperature where decomposition, exothermal reaction and ignition occur spontaneously. This critical temperature range depends upon the chemical composition of polymer.

CHAPTER 3

METHODOLOGY

3.1 RESEARCH METHODOLOGY

3.1.1 Methodology for FYP 1

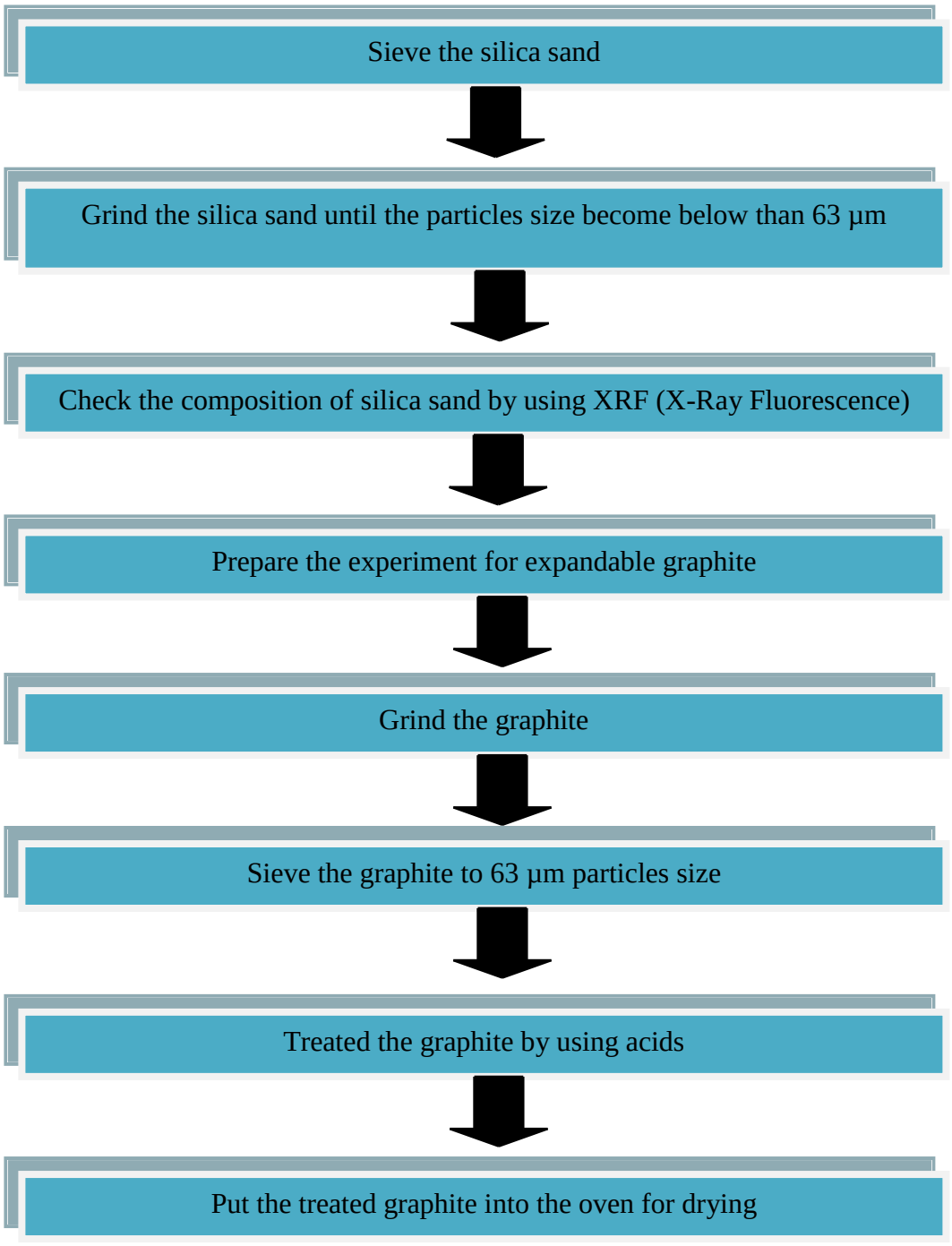


Figure 1: Methodology for FYP 1

3.1.2 Methodology for FYP 2

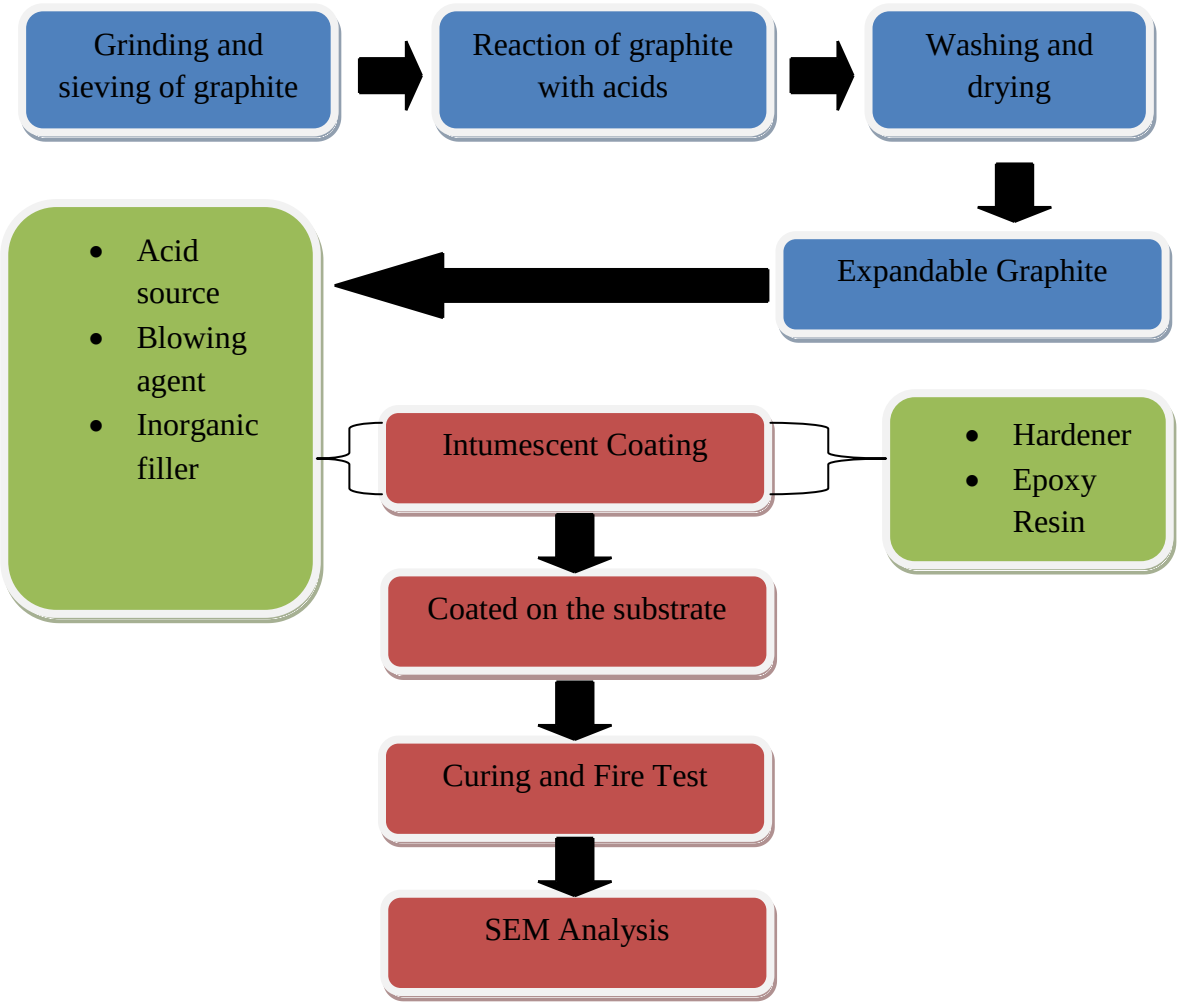


Figure 2: Methodology for FYP 2

3.2 PROJECT PLANNING

3.2.1 Gantt Chart FYP 1

No.	Action/Plan	WEEK													
		1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Selection of Project Topic								M							
2	Preliminary Research Work								I							
3	Submission of Preliminary Report								D							
4	Research/ Literature Review															
5	Preparation of silica sand: • Sieve The Silica Sand • Grind the silica sand • Analyze the composition of silica sand								S E M B							
6	Preparation of expandable graphite: • Grind the graphite • Sieve the graphite • Treated the graphite								R E A K							
7	Submission of Progress Report															
8	Seminar															

9	Analyze The Graphite															
10	Interim Report Preparation															
11	Submission of Interim Report Final Draft															
12	Oral Presentation															

Table 1: Gantt Chart for FYP 1

3.2.2 Gantt Chart FYP 2

No.	Action/Plan	WEEK													
		1	2	3	4	5	6	7	M	8	9	10	11	12	13	14
1	Preparation of coating • Expandable Graphite								I							
2	Fire Test and coating analysis with SEM								D							
3	Submission of Progress Report 1								S							
4	Project work continue								E							
5	• Submission of Progress Report 2								M							
6	Seminar								B							
									R							

7	Preparation of coating • Expandable Graphite with silica sand								E A K								
8	Fire Test and coating analysis with SEM																
9	Poster Exhibition																
10	Submission of dissertation (final draft)																
11	Oral presentation																
12	Submission of dissertation (hard bound)																

Table 2: Gantt Chart for FYP 2

3.3 MATERIALS, EQUIPMENTS AND TOOLS REQUIRED

3.3.1 Material Used

- Silica Sand (Filler).
- Expandable Graphite.
- Ammonium Polyphosphate (APP).
- Bisphenol A (BPA)
- Triethylenetetramine (TETA).
- Melamine (MEL).
- Boric Acid.
- Sulphuric Acid
- Acetic Acid

3.3.2 Type of tools

- Oven is used to dry the wet silica sand at 60^0C for 1 day.
- Ball Mill is used to grind the silica sand until the particles size become lower than 63 µm.
- Grinder is used to grind the graphite.
- Sieve Shaker is use to sieve the graphite in order to get the 63 µm particles size of graphite.
- Furnace is use to burn the coating sample at 500^0C for 2 hours.
- Bunsen Burner is use to burn the coating sample for Fire Test.
- XRF (X-Ray Fluorescence) to check and determine the chemical composition of silica sand.
- SEM (Scanning Electron Microscopy) to check the microstructure of the coating for outer surface and inner surface of char layer after Fire Test.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 PREPARATION OF SILICA SAND TO BELOW THAN µm PARTICLES SIZE

1) The silica sand had been put in the oven for 1 day at 60^0C to dry it.
2) The silica sand had been grinded by using Ball Mill. It takes 3 weeks to complete the grinding process until the particles size of silica sand become less than 63 µm.
3) After grinding, the composition of silica sand had been checked by using X-Ray Fluorescence (XRF).

Figure 3: Silica sand had been put into the oven

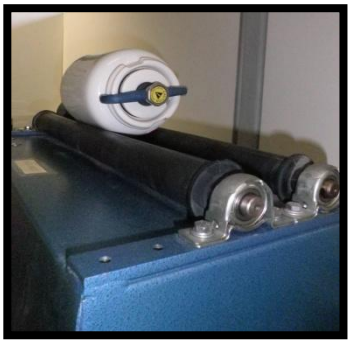


Figure 4: Silica sand had been grinded by using Ball Mill

Figure 5: BEFORE; Particles size: Micron meter, µm

Figure 6: AFTER; Particles size: grinded sand below than micron meter, µm

4.2 PREPARATION OF EXPANDABLE GRAPHITE

Before preparing for expandable graphite, the graphite had been grinded first by using grinder. Next, the graphite had been sieved to 63 µm particles size. The graphite had been treated based on the formulations as followed:

No	Graphite (%)	Acetic Acid (%)	Sulphuric Acid (%)	Potassium Permanganate (%)
1	28.0	56.0	14.0	1.96
2	28.0	56.0	14.0	1.96
3	28.0	56.0	14.0	1.96

Table 3: Formulations for Expandable Graphite

Equipment used:

- Hot Plate, pipette, dropper, thermometer, beaker, magnet, condenser, spatula, three necks round bottom flask, bowl, small container, small tube.

Materials used:

- Graphite, Acetic Acid (CH_3COOH), Sulphuric Acid (H_2SO_4), Potassium Permanganate ($KMnO_4$), distilled water

Procedure:

1) The equipment set up had been made as followed:

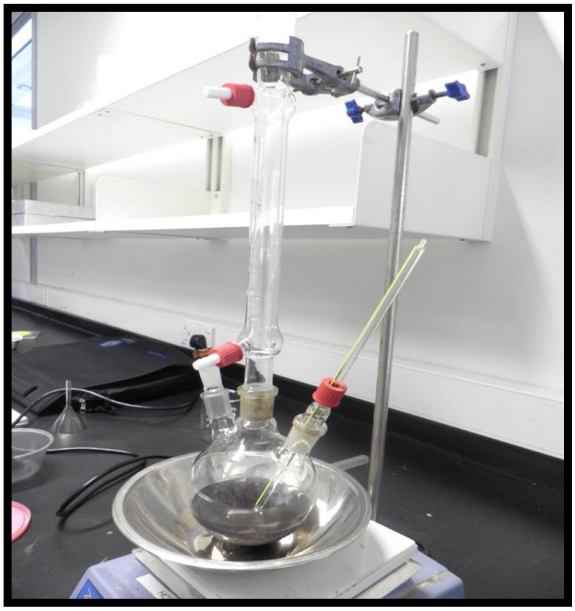


Figure 7: Equipments set up for treated graphite experiment

2) The graphite had been prepared and the graphite had been poured into the smaller container.
3) The Potassium Permanganate had been mixed with the graphite in the small container.
4) The mixture had been stirred by using spatula.
5) The mixture had been poured into the three necks round bottom flask.
6) The magnet had been put into the three necks round bottom flask and mixed well.
7) Then, the Acetic Acid had been poured into the flask.
8) The temperature of the mixture had been checked by using thermometer and maintained at room temperature.
9) The small tube had been connected through the pipe into the top hole of the condenser and the other tube is connected to the sink from the bottom hole of the condenser.

10) The pipe had been opened to make the water flow through the condenser and maintained at room temperature (25^0C).
11) The speed to stir the mixture is 2.5 rpm (revolution per minutes).
12) The Sulphuric Acid had been slowly dropped by using dropper into the three necks round bottom flask.
13) The process had been maintained for one hour.
14) After 1 hour, the mixture had been washed by using distilled water.
15) Lastly, the mixture had been put into the oven for 3 hours at 60^0C to dry it.

Preparation for Expandable Graphite

Figure 8: Graphite before it been grinded

Figure 9: The graphite had been grinded by using grinder

Figure 10: The grinder had been tightened to prevent the waste of graphite	*Figure 11: Graphite after grinded*
	
Figure 12: Graphite had been prepared for sieving process	*Figure 13: Graphite had been seived by using Sieve Shaker to get 63* µm particles size
	
Figure 14: Equipment preparation for graphite treatment	*Figure 15:The graphite had been treated by using sulphuric acid and acetic acid. The mixture had been washed by using distilled water and had been put into the oven for drying process*

4.3 INTUMESCENT COATING WITH EXPANDABLE GRAPHITE (EG)

4.3.1 Formulations and procedure of the intumescent coating sample with EG

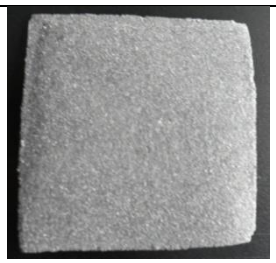
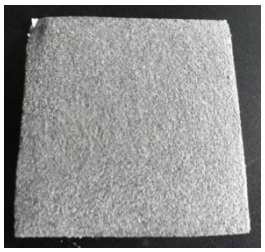
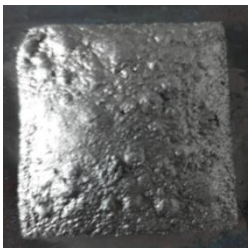
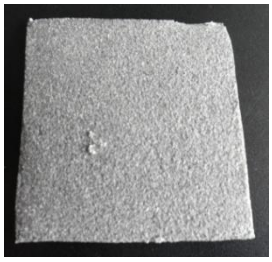
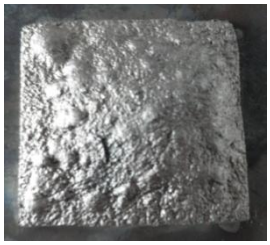
The intumscent coating with expandable graphite had been done by using 14 formulations for 63 µm particles size of expandable graphite.

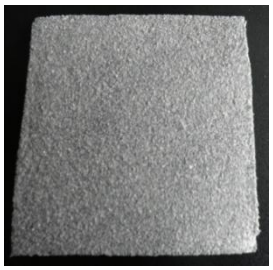
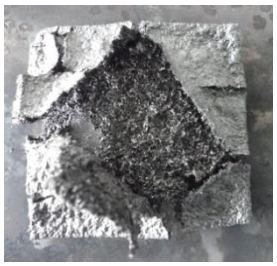
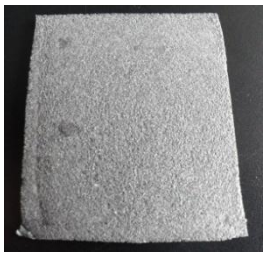
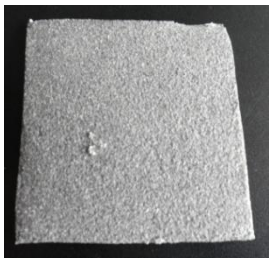
No	Expandable Graphite (%)	Ammonium Polyphosphate, APP (%)	Melamine (%)	Boric Acid (%)	Epoxy (%)	Hardener (%)
DT01	0.00	11.11	11.11	5.56	44.44	22.22
DT02	5.56	11.11	11.11	5.56	44.44	22.22
DT03	5.56	11.11	8.33	8.33	44.44	22.22
DT04	5.56	11.11	5.56	11.11	44.44	22.22
DT05	5.56	8.33	11.11	8.33	44.44	22.22
DT06	5.56	5.56	11.11	11.11	44.44	22.22
DT07	8.33	11.11	8.33	5.56	44.44	22.22
DT08	11.11	11.11	5.56	5.56	44.44	22.22
DT09	8.33	8.33	11.11	5.56	44.44	22.22
DT10	11.11	5.56	11.11	5.56	44.44	22.22
DT11	8.33	8.33	8.33	8.33	44.44	22.22
DT12	5.56	8.33	8.33	11.11	44.44	22.22
DT13	11.11	8.33	8.33	5.56	44.44	22.22
DT14	8.33	11.11	5.56	8.33	44.44	22.22

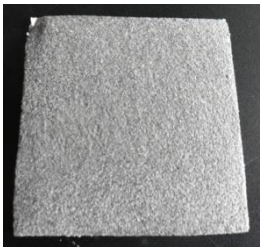
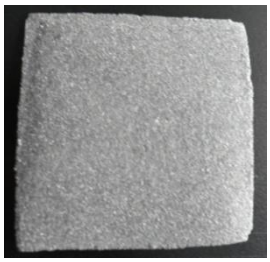
Table 4: Formulations for Intumescent Coating with Expandable Graphite

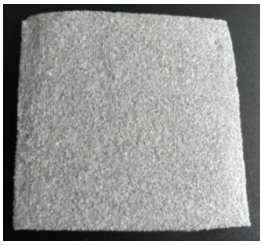
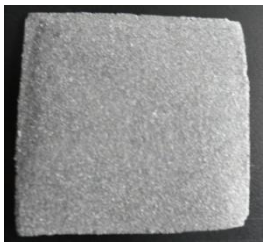
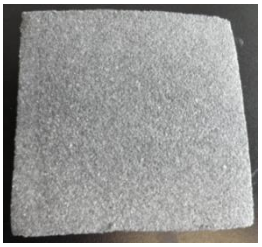
The entire coating sample for each formulation had been put into the furnace for fire test at 450^0C for one hour. Table 2 shows the picture and the thickness of the coating before and after fire test.

No	Before Fire Test	After Fire Test
DT01	 Thickness of steel substrate= 1.50mm Thickness of the coating = 1.90mm	 Thickness of the char = 1.98mm (1.04 times expansion) • Rough surface, very little cracks and not detachable.
DT02	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.15mm	 Thickness of the char = 4.50mm (2.09 times expansion) • Rough surface, many cracks and not detachable.
DT03		

	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.86mm	 Thickness of the char = 5.50mm (1.92 times expansion) • Smooth surface, many cracks and not detachable.
DT04	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.67mm	 Thickness of the char = 8.50mm (5.83 times expansion) • Very smooth surface, no cracks and not detachable.
DT05	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.26mm	 Thickness of the char = 7.50mm (3.32 times expansion) • Very smooth surface, no cracks and not detachable.

DT06	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.76mm	 Thickness of the char = 2.90mm (1.05 times expansion) • Very rough surface, a lot of cracks and detachable.
DT07	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.77mm	 Thickness of the char = 3.50mm (1.26 times expansion) • Rough surface with a little cracks and not detachable.
DT08	 Thickness of steel substrate= 1.50mm	 Thickness of the char = 6.50mm

	Thickness of the coating = 2.99mm	(2.17 times expansion) • Very smooth surface with a little cracks and not detachable.
DT09	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.70mm	 Thickness of the char= 5.50mm (2.04 times expansion) • Very rough surface with many cracks and detachable.
DT10	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.90mm	 Thickness of the char= 3.00mm (0.10 times expansion) • Smooth surface with small cracks and detachable.
DT11		 Thickness of the char = 6.50mm

	Thickness of steel substrate= 1.50mm Thickness of the coating = 2.94mm	(2.21 times expansion) • Very smooth surface, a little cracks and not detachable.
DT12	 Thickness of steel substrate= 1.50mm Thickness of the coating = 3.20mm	 Thickness of the char = 3.60mm (1.13 times expansion) • Very rough surface, big cracks and not detachable.
DT13	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.06mm	 Thickness of the char = 2.32mm (1.13 times expansion) • Very rough surface, big cracks and detachable.
DT14		

	Thickness of steel substrate= 1.50mm Thickness of the coating = 2.70mm	Thickness of the char= 2.85mm (1.06 times expansion) • Very rough surface, many cracks and not detachable.

Table 5: the thickness of the Coating Sample with Expandable Graphite before and after Fire Test by using Furnace

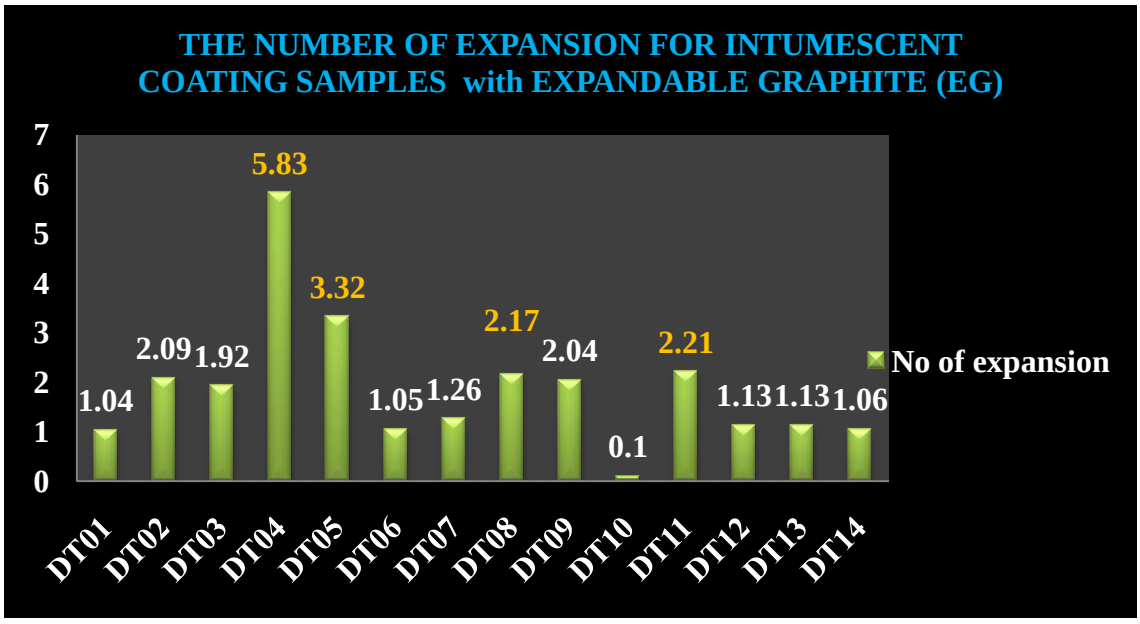


Figure 16: Graph the Number of Expansion for Intumescent Coating Samples with EG

Based on Figure 16, there are 4 samples which are DT04, DT05, DT08 and DT11 give greater expansion compare to others. The greater expansion is 5.83 times of expansion for DT04 compared to others. This is because the char layer expansion of the coating for the 4 samples is higher and the surface of the coating is very smooth with little cracks and not detachable. This char layer which is the insulating layer will protect the steel structure during fire. The microstructure for these samples had been examined by using Scanning Electron Microscopy (SEM).

4.3.2 Microstructure Characterization

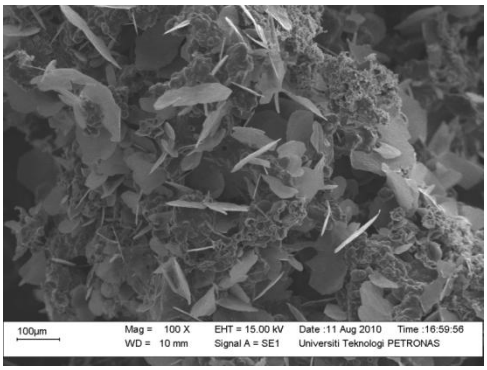


Figure 17: SEM Micrograph of DT04 coating with expandable graphite (inner surface): 100X

Figure17 show the microstructure of the coating with expandable graphite. The coating is good and illustrates the intumescent behavior. The emission of N_2, NH_3 and CO_2 gas and dehydration of water occurred. The graphite flakes appear in the inner surface and produce the heat barrier to protect the steel (substrate).

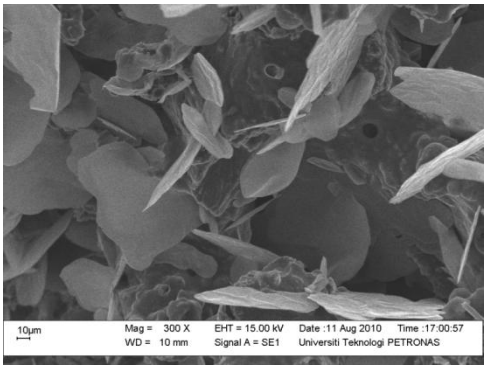


Figure 18: SEM Micrograph of DT04 coating with expandable (inner surface): 300X

Figure 18 show the microstructure of the coating with expandable graphite. The flakes are very thick and illustrate the heat barrier of the coating to protect the substrate from fire.

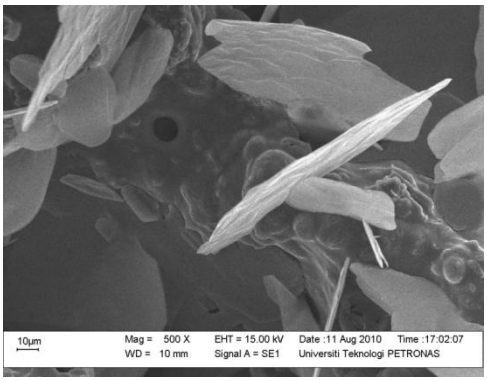


Figure 19: SEM Micrograph of DT04 coating of expandable graphite with boric acid (inner surface): 500X

Figure 19 show the microstructure of the coating with expandable graphite burnt at 450^0C . There are small holes, bubbles and thick flakes occurred on the coating surface due to dehydration of water and emission of N_2, NH_3 and CO_2 gas.

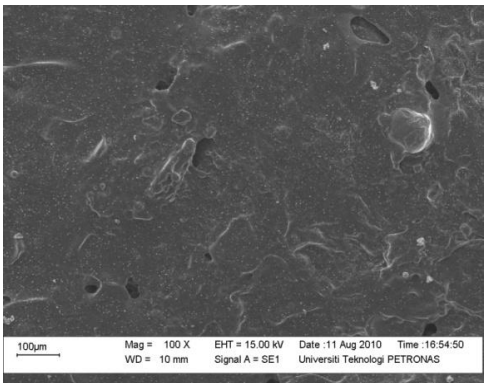


Figure 20: SEM Micrograph of DT04 coating with expandable graphite (outer surface): 100X

Figure 20 show the microstructure of the coating with expandable graphite burnt at 450^0C . The outer surface of the coating is smooth. The small holes appeared where the heat can dissipated from the hole and prevent the heat transfer to the surface. This is due to dehydration of water and emission of N_2, NH_3 and CO_2 gas.

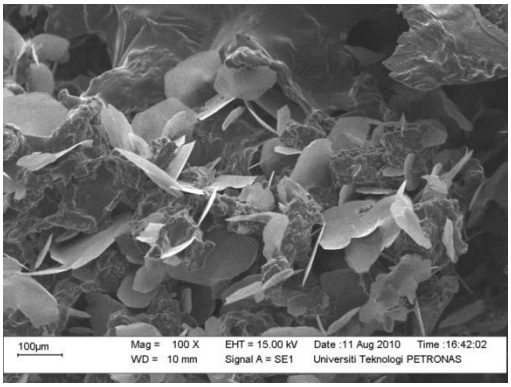


Figure 21: SEM Micrograph of DT05 coating with expandable graphite (inner surface): 100X

Figure 21 show the microstructure of the coating with expandable graphite burnt at 450^0C . The inner surface of the char is very crispy and a lot of flakes appeared due to dehydration of water.

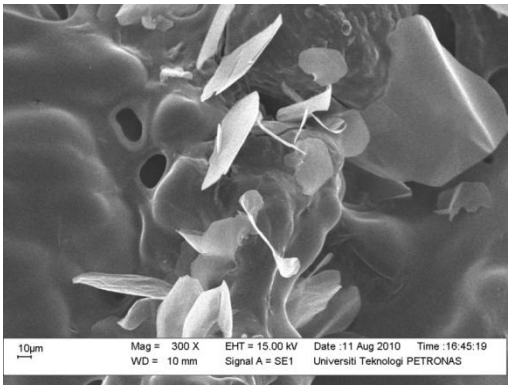


Figure 22: SEM Micrograph of DT05 coating with expandable graphite (inner surface): 300X

Figure 22 show the microstructure of the coating with expandable graphite burnt at 450^0C . There are bubbles and flakes and the thin layer of the bubbles breakdown and some holes appeared due to dehydration of N_2, NH_3 and CO_2 gas.

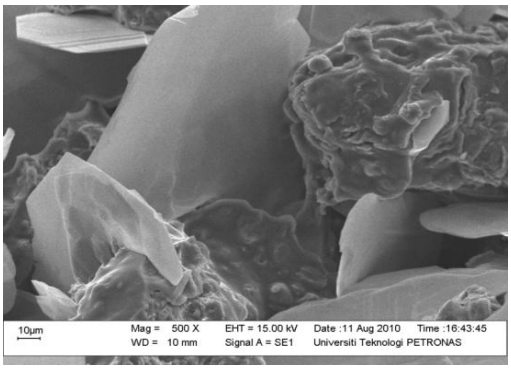


Figure 23: SEM Micrograph of DT05 coating with expandable graphite (inner surface): 500X

Figure 23 show the microstructure of the coating with expandable graphite burnt at 450^0C . The thick layer of the char and flakes appeared in the inner surface due to dehydration of water and emission of N_2, NH_3 and CO_2 gas.

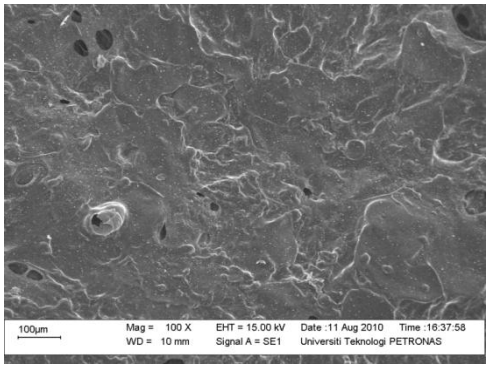


Figure 24: SEM Micrograph of DT05 coating with expandable graphite (outer surface): 100X

Figure 24 show the microstructure of the coating with expandable graphite burnt at 450^0C . The outer surface of the coating is smooth and small holes appeared because no cracks appeared on the coating surface.

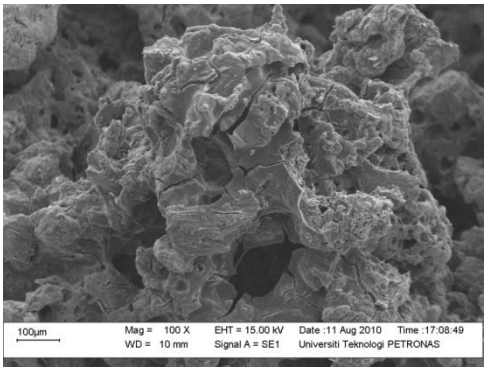


Figure 25: SEM Micrograph of DT08 coating with expandable graphite (inner surface): 100X

Figure 25 show the microstructure of the coating with expandable graphite burnt at 450^0C . There are cracks in the inner surface and the coating swell nicely due to dehydration of water and emission of gas from the holes.

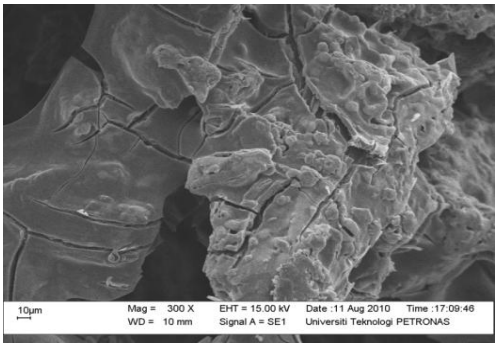


Figure 26: SEM Micrograph of DT08 coating with expandable graphite (inner surface): 300X

Figure 26 show the microstructure of the coating of expandable graphite with boric acid burning at 450^0C . The cracks appeared on the char. The char swelled due to the emission of gas.

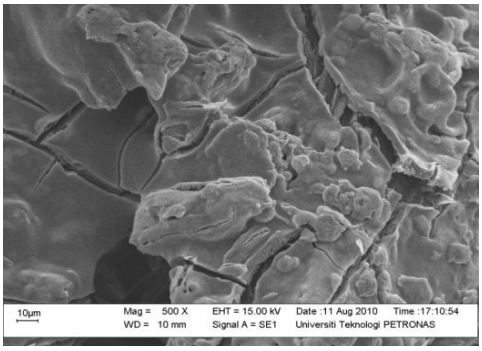


Figure 27: SEM Micrograph of DT08 coating with expandable graphite (inner surface): 500X

Figure 27 show the microstructure of the coating with expandable graphite burnt at 450^0C . There are also cracks appeared in the inner surface of the char due to dehydration of water and emission of gas.

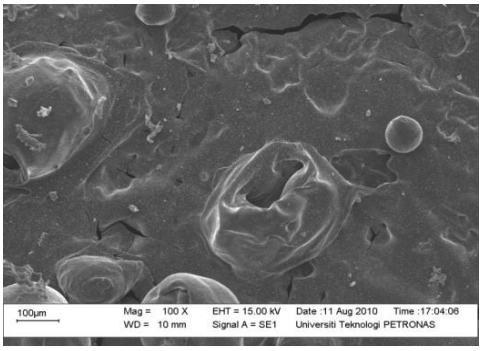


Figure 28: SEM Micrograph of DT08 coating with expandable graphite (outer surface): 100X

Figure 28 show the microstructure of the coating with expandable graphite burnt at 450^0C. There are bubbles which is representing the emission of gas. The outer surface of the char layer not very smooth. This is due to a lot of cracks appeared on the coating surface.

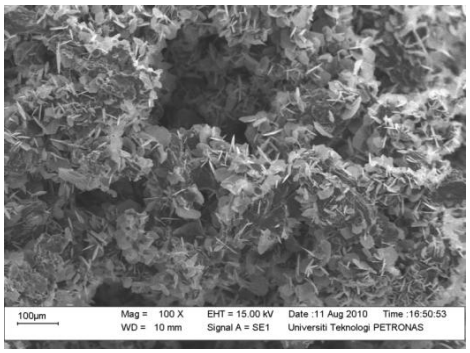


Figure 29: SEM Micrograph of DT11 coating with expandable graphite (inner surface): 100X

Figure 29 show the microstructure of the coating with expandable graphite burnt at 450^0C . The inner surface of the coating is very good and no bubbles. A lot of flakes appear and no heat transfer between the surfaces due to emission of N_2, NH_3 and CO_2 gas and dehydration of water.

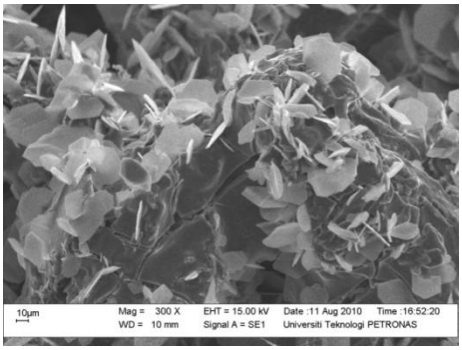


Figure 30: SEM Micrograph of DT11 coating with expandable graphite (inner surface): 300X

Figure 30 show the microstructure of the coating with expandable graphite burnt at 450^0C . The inner surface of the coating is very nice. A lot of flakes appear and the char is very crispy due to dehydration of water.

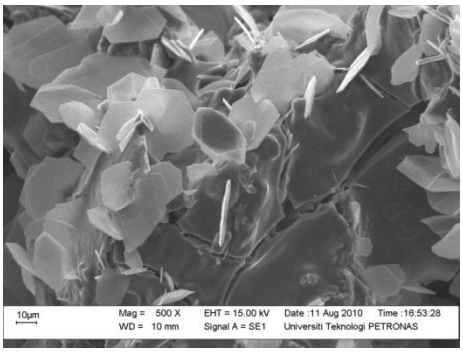


Figure 31: SEM Micrograph of DT11 coating with expandable graphite (inner surface): 500X

Figure 31 show the microstructure of the coating with expandable graphite burnt at 450^0C . A lot of flakes appeared and produce the heat barrier between the surfaces to protect the steel (substrate). There is also small crack in the inner surface of the coating due to dehydration of water and emission of gas.

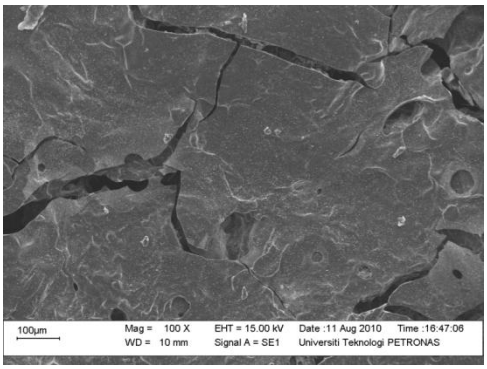


Figure 32: SEM Micrograph of DT11 coating with expandable graphite (outer surface): 100X

Figure 32 show the microstructure of the coating with expandable graphite burnt at 450^0C . The cracks appeared in the outer surface of the coating. No bubbles and the coating are very smooth due to the emission of gas.

4.4 INTUMESCENT COATING WITH EXPANDABLE GRAPHITE (EG) AND SILICA SAND

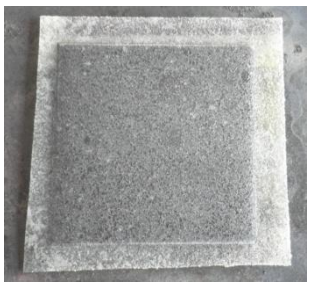
4.4.1 Formulations and procedure of the intumescent coating sample with EG and Silica Sand

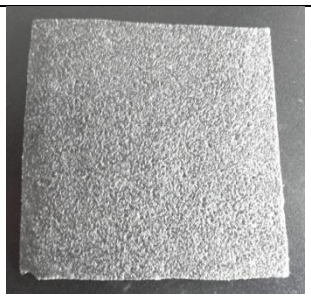
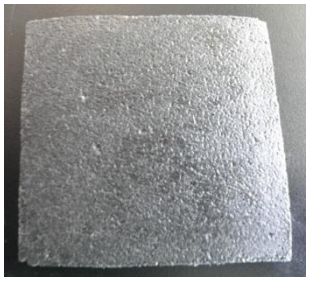
The intumscent coating of expandable graphite with silica sand had been done by using 8 formulations for 63 µm particles size of expandable graphite.

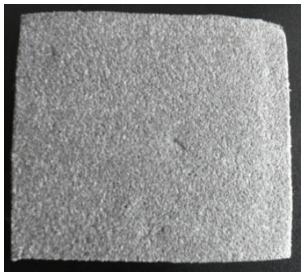
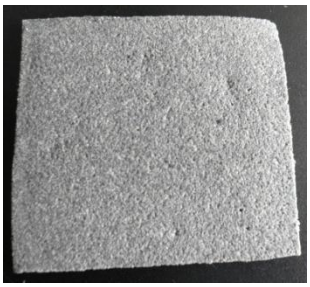
No	EG (%)	APP (%)	MEL (%)	Boric Acid (%)	Silica Sand (%)	Epoxy (%)	Hardener (%)
DS01	-	-	-	-	-	66.67	33.33
DS02	-	-	-	-	33.33	44.44	22.22
DS03	16.67	-	-	-	16.67	44.44	22.22
DS04	13.89	12.50	-	-	6.94	44.44	22.22
DS05	13.89	27.78	13.89	-	27.78	11.11	5.56
DS06	13.89	27.78	13.89	19.44	8.33	11.11	5.56
DS07	13.89	27.78	13.89	16.67	11.11	11.11	5.56
DS08	13.89	27.78	13.89	13.89	13.89	11.11	5.56

Table 6: Formulations for Intumescent Coating Expandable Graphite with Silica Sand

The entire coating sample for each formulation had been put into the furnace for fire test at 450^0C for one hour. Table 2 shows the picture and the thickness of the coating before and after fire test.

No	Before Fire Test	After Fire Test
DS01	 Thickness of steel substrate= 1.50mm Thickness of the coating = 0.51mm	 Thickness of the char = 0.54mm (0.03 times expansion) • Rough surface and detachable
DS02	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.85mm	 Thickness of the char = 3.20mm (1.12 times expansion) • Rough surface, many cracks and detachable from substrate
DS03		

	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.75mm	 Thickness of the char = 2.96mm (1.08 times expansion) • Rough surface, many cracks and detachable from substrate
DS04	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.67mm	 Thickness of the char = 46.50mm (17.42 times expansion) • Very smooth surface, no cracks and not detachable.
DS05	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.50mm	 Thickness of the char = 23.50mm (9.40 times expansion)

		• Very smooth surface, no cracks and not detachable.
DS06	 Thickness of steel substrate= 1.50mm Thickness of the coating = 3.31mm	 Thickness of the char = 13.50mm (4.08 times expansion) • Smooth surface, very little cracks and not detachable.
DS07	 Thickness of steel substrate= 1.50mm Thickness of the coating = 3.98mm	 Thickness of the char = 9.50mm (2.39 times expansion) • Smooth surface with little cracks and not detachable.
DS08		

	Thickness of steel substrate= 1.50mm Thickness of the coating = 3.47mm	Thickness of the char = 3.80mm (0.33 times expansion) • Very rough surface with many cracks and detachable.

Table 7: the thickness of the Coating Sample with Expandable Graphite and Silica Sand before and after Fire Test by using Furnace

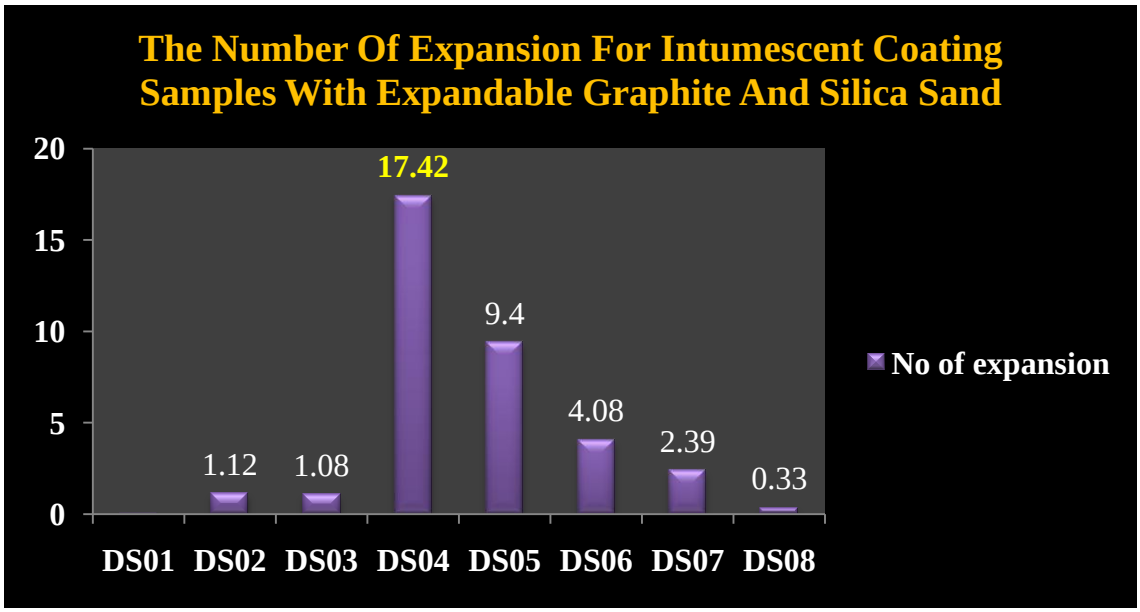


Figure 33: Graph the Number of Expansion for Intumescent Coating Samples with Expandable Graphite and Silica Sand

Based on Figure 33, the graph shown the number of expansion for intumescent coating samples with expandable grahite and silica sand. DS04 sample has the greater expansion of char layer which 17.42 times of expansion compared to others. This is because, by adding silica sand as filler into the intumescent coating, the coating will swell and expand. It will create an insulating barrier to protect the char coating. The lowest expansion is DS08 samples which are 0.33 times of expansion.

4.4.2 Microstructure Characterization

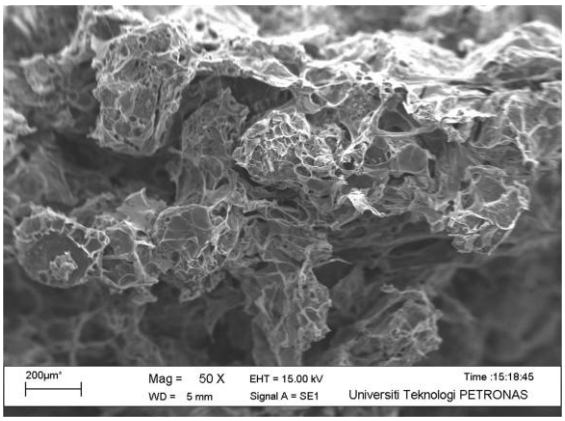


Figure 34: SEM Micrograph of DS04 coating with expandable graphite and silica sand (inner surface): 50X

Figure 34 show the microstructure of the coating with expandable graphite and silica sand burnt at 450^0C . The cracks appeared in the outer surface of the coating. No bubbles and the coating are very smooth due to the emission of gas.

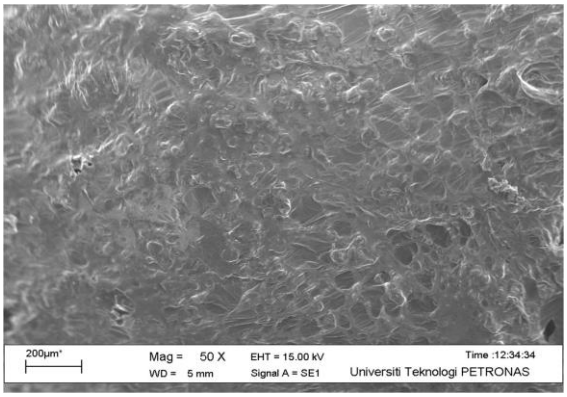


Figure 35: SEM Micrograph of DS04 coating with expandable graphite and silica sand (outer surface): 50X

Figure 35 show the microstructure of the coating with expandable graphite and silica sand burnt at 450^0C . There are a lot of bubbles appeared on the outer surface of the char coating. This is due to the emission of gases. A lot of bubbles are better because the gas produce will expand the char.

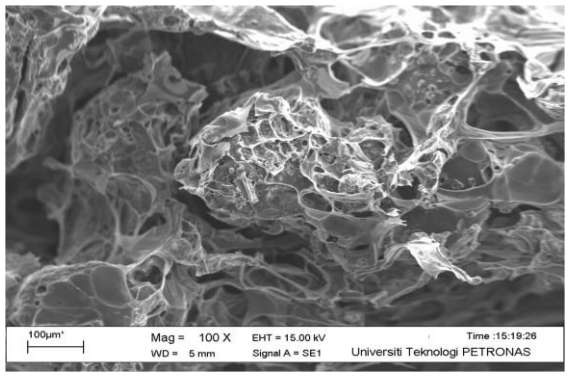


Figure 36: SEM Micrograph of DS04 coating with expandable graphite and silica sand (inner surface): 100X

Figure 36show the microstructure of the coating with expandable graphite and silica sand burnt at 450^0C . There are cracks appeared on the outer surface of the char coating. This is due to the hydration of water. Less cracks on the inner surface is more better to prevent the heat penetrate the substrate.

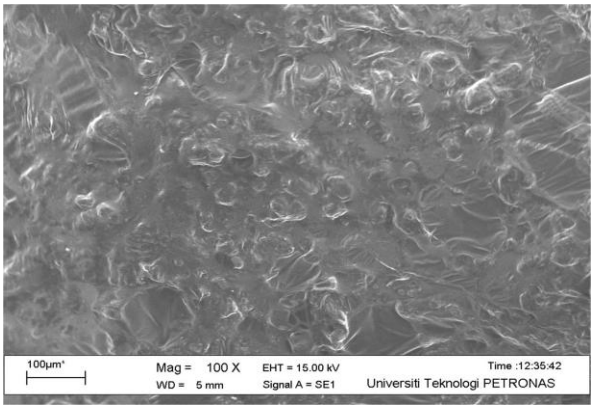


Figure 37: SEM Micrograph of DS04 coating with expandable graphite and silica sand (outer surface): 100X

Figure 37 show the microstructure of the coating with expandable graphite and silica sand burnt at 450^0C . There are a lot of bubbles appeared on the outer surface of the char layer due to the emission of gases and cause the coating to expand.

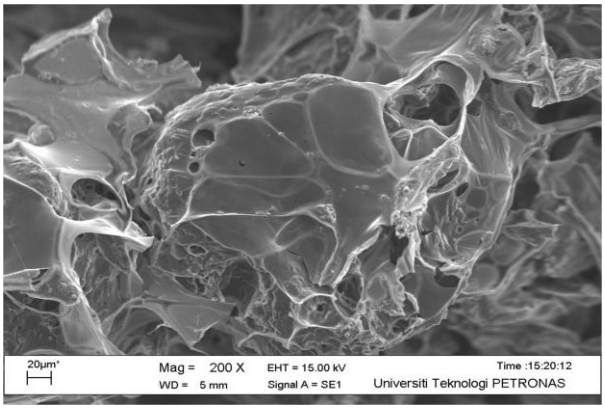


Figure 38: SEM Micrograph of DS04 coating with expandable graphite and silica sand (inner surface): 200X

Figure 38 show the microstructure of the coating with expandable graphite and silica sand burnt at 450^0C . There are cracks appeared on the outer surface of the char coating. This is due to the hydration of water. Less cracks on the inner surface is more better to prevent the heat penetrate the substrate.

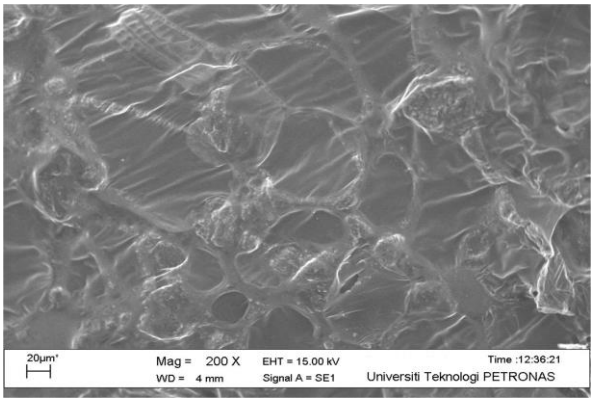
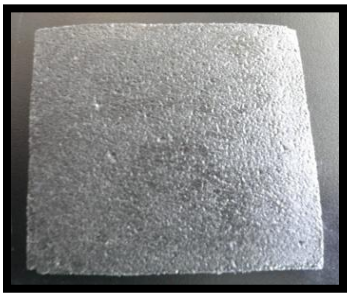
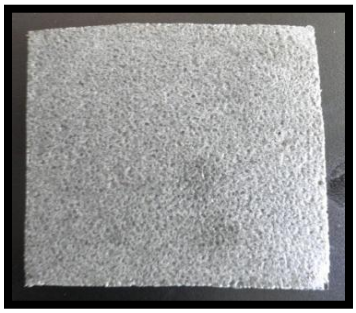
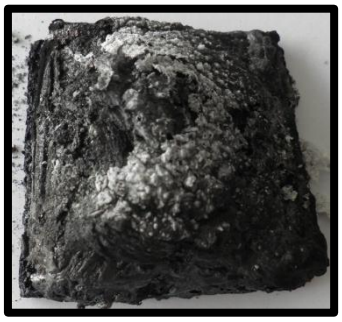


Figure 39: SEM Micrograph of DS04 coating with expandable graphite and silica sand (outer surface): 200X

Figure 39 show the microstructure of the coating with expandable graphite and silica sand burnt at 450^0C . There are a lot of bubbles appeared on the outer surface of the char coating due to the emission of gases from blowing agent and cause the char to expand. More bubbles is more better because the char will expand more and insulate the substrate

4.4.3 Fire Test for the intumescent coating sample with Expandable Graphite And Silica sand by using Bunsen Burner

No	Before Fire Test	After Fire Test
DS04	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.67mm	 Thickness of the char = 40.00mm (15 times of expansion) • Very smooth surface, no cracks and not detachable.
DS05	 Thickness of steel substrate= 1.50mm Thickness of the coating = 2.50mm	 Thickness of the char = 20.00mm (8 times of expansion) • Very smooth surface, no cracks and not detachable.
DS06		

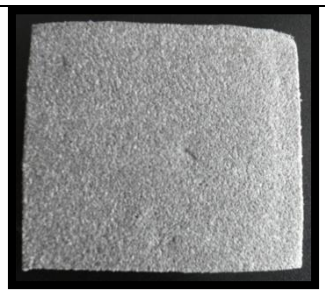
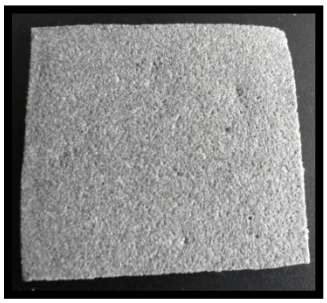
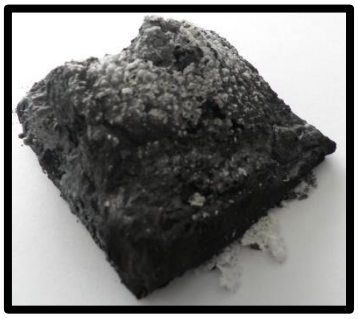
	 Thickness of steel substrate= 1.50mm Thickness of the coating = 3.31mm	 Thickness of the char = 11.45mm (3.46 times of expansion) • Smooth surface, very little cracks and not detachable.
DS07	 Thickness of steel substrate= 1.50mm Thickness of the coating = 3.98mm	 Thickness of the char = 8.20mm (2.06 times expansion) • Smooth surface cracks and not detachable from steel substrate.

Table 8: the thickness of the Coating Sample with Expandable Graphite before and after Fire Test by using Bunsen Burner

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

Intumescent coatings are thin film coatings which when applied under heat of fire and due to endothermic reactions, the coating expands or foams and creates a carbonaceous char coating on the steel which then solidifies. The char layer will protect the substrate. Intumescent coating contains four basic elements which are acid source (APP), carbon source (graphite, PER), blowing agent (melamine) and binder (epoxy resin). The silica sand had been prepared by sieving the sand using Sieve Shaker. Then, the silica sand had been grinded by using Ball Mill until the particles size become smaller less than µm. The compositions of the silica sand had been analyzed by using XRF (X-Ray Fluorescence) testing. Graphite had been grinded by using grinder and sieved by using Sieve Shaker to 63 µm particles size by using Sieve Shaker. Treatment of graphite to convert to expandable graphite had been done by using sulphuric acid and acetic acid. The intumscent coating with expandable graphite had been done by using 14 formulations for 63 µm particles size of expandable graphite. The Fire Test had been done by using furnace. From 14 formulations, only 4 formulations which are DT04, DT05, DT08 and DT11 give greater expansion. This is because the char layer expansion of the coating for the 4 formulations is higher and the surface of the coating is very smooth with little cracks and not detachable. This char layer which is an insulating layer will protect the steel structure during fire. The microstructure for these formulations had been examined by using Scanning Electron Microscopy (SEM). The intumscent coating with expandable graphite and silica sand had been done by using 8 formulations for 63 µm particles size of expandable graphite. The Fire Test had been done by using furnace and Bunsen Burner. Among 8 formulations, a DS04 formulation has the greater expansion of char layer which is 17.42 times of expansion compared to others. This is because, by adding silica sand as filler into the intumescent coating, the coating will swell and expand. It will create an insulating barrier to protect the char coating. The lowest expansion is DS08 formulations which are 0.33 times of expansion. The

microstructure for these formulations had been characterized by using Scanning Electron Microscopy (SEM). The coating was well bounded with substrate during Fire Test. The addition of silica sand creates an insulating barrier to protect char coating. Thus, reducing the heat transfer to the substrate and improving the efficiency of intumescent coating.

5.2 RECOMMENDATIONS

In this project, the silica sand had been grinded by using Ball Mill. It is recommended to grind the silica sand by using grinder to get the lower particles size of silica sand. By using grinder, the time taken to produce the grinded silica sand is less compared by using Ball Mill. The lower the particles size, the greater the char expansion.

REFERENCES

[1] Corrosion Science 49 (2007) 2237-2253, *Influence of Expendable Graphite on Fire Resistance and Water Resistance of Flame-Retardant Coatings, Zhengyu Wang, Enhou Han and Wei Ke.*

[2] Polymer Degradation and Stability 91 (2006) 1937-1947, *Effects of Acrylic Polymer and Nanocomposite with Nano-SiO_2 on Thermal Degradation and Fire Resistance of APP-DPER-MEL Coating.*

[3] Fire Technology August 1989, *Soluble Silicate Based Coatings for Fire Protection by D.E.Vinot, K.B.Langille, D.Nguyen and J.O.Bernt,* Springer Netherlands.

[4] European Coatings 6/2007, *New Developments in Intumescent Paints for Structural Steel Fire Protection by T.Futterer, D.Garcia, V.Mans, H-D.Nagerl, E.Tortosa by Zhengyu Wang, Enhou Han and Wei Ke.*

[5] Journal Article Excerpt, *Brief Review of Intumescent Fire Retardant Coatings by J.Hao, W.K.Chow.*

[6] Journal Article Dec 19, 1989, *Inorganic Intumescent Fire Protective Coatings by Dat T.Nguyen, Dwight E.Veinot, and James Foster.*

[7] Fire Safety-'90; SURVIAC 18708; *Intumescent Coating, Low Cost Foam Insulation, and Used of Gel Water for Fire Protection and Suppression; Nov1990 by William A.Mallow and Patrick H.Zabel.*

[8] Journal Article Dec 18, 1979, *Method of Forming Fire Resistant Silicate Coating by Reinhard Kalbskopf and Felix Trojer.*

[9] Journal Article 1976, *Behavior of Expandable Graphite as Flame Retardant in Flexible Polyurethane Foam by D.W.Krassowski.*